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Cryogenics and Space Technology*

[Cryogénie et technologie spatiale]

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SOMMAIRE. Le Laboratoire de Cryogénie du »National Bureau of Standards« projette plusieurs expériences relatives au programme spatial des Etats-Unis. La plupart de celles-ci résultent de la décision d'utiliser de l'hydrogène liquide comme agent de propulsion des fusées. Parmi les plus intéressantes de ces expériences, on peut citer:

Mesure des propriétés du parahydrogène à l'état liquide et à l'état gazeux entre 15° et 100°K à des pressions atteignant 340 atm. Ces mesures comprennent les relations p-v-t, la chaleur spécifique, la conductibilité thermique, la viscosité, la vitesse du son et la constante diélectrique. Des tableaux et des diagrammes des propriétés thermodynamiques dérivées sont en cours de préparation.

Un type de projet très différent est l'étude des difficultés rencontrées lors de l'essai de refoulement d'une vapeur sèche provenant d'un réservoir d'hydrogène liquide de propulsion en l'absence d'un champ de gravitation pour effectuer la séparation de phase. Il est apparu qu'un séparateur centrifuge conviendrait, mais cela a entraîné un autre problème: celui des paliers qui fonctionneront dans l'hydrogène liquide ou gazeux à des températures voisines du point d'ébullition de l'hydrogène.

Comme il sera nécessaire de refouler l'hydrogène dans le vide spatial on craignait que les gouttelettes de liquide entraînées ne vinssent à se congeler et à boucher la conduite. Une étude de laboratoire a montré que cette crainte était justifiée.

When liquid parahydrogen was selected as a propellant for some of the more advanced space vehicles and rocket stages, it became apparent that much of the information needed by the design engineers was either inadequate or non-existent. The many properties of parahydrogen were not precisely known, or known only in very limited regions of pressure. Experimental work was needed to predict the behavior of liquid and cold gaseous hydrogen in the tanks, pumps, valves and instruments of the rocket system. New special purpose instruments were needed. The properties of many materials of construction were not known in this temperature region. The behavior of moving mechanisms could not be predicted.

Because of these circumstances, the National Bureau of Standards Cryogenic Engineering Laboratory, with support from the National Aeronautics and Space Administration, undertook several experimental projects specifically designed to provide some of the more urgently needed information.

PROPERTIES OF PARAHYDROGEN

A. *Pressure-Volume-Temperature Relations*

Figure 1 is a cross section of the piezometer region of the PVT cryostat [1]. The sample is contained in the thick-walled, 25 ml copper pipet, which can be cooled by admitting hydrogen into the reflux tube so that condensation on the upper part and evaporation at the pipet transfers heat from the pipet to the H_2 tank. The temperature of the sample is measured with a strain-free, capsule-type platinum resistance thermometer. The pressure is transmitted through the capillary tube to pressure measuring equipment consisting of a precision piston-type pressure balance. The hydrogen is isolated from the oil in the pressure balance by a sensitive diaphragm fitted with electrical contacts to in-

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dicate the null position. The experimental procedure consists of filling the pipet with parahydrogen at the desired starting temperature and pressure, adjusting the tempera-

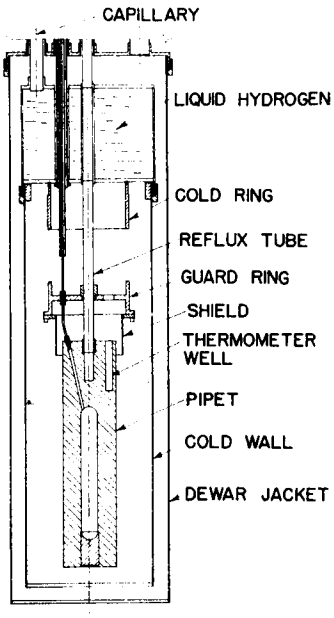

Fig. 1. The P-V-T Cryostat

ture to an exact integral Kelvin temperature, and reading the pressure. Next the temperature is raised to a higher integral temperature and the pressure again read. This is continued until a pressure of about 320 atm or a temperature of 100°K is reached. After completing such a set of measurements, the amount of hydrogen in the pipet is measured by releasing it into a gasometer system consisting of a set of calibrated spherical glass bulbs and a precision manometer. Both the bulbs and manometer are maintained at constant, uniform temperatures.

Next, the pipet is filled to a different density and another set of measurements made. Each such set of measurements constitutes a pseudo-isochore since the density remains constant except for the small decrease caused by the slight amount of fluid that flows into the capillary and diaphragm cell as the pressure rises.

To obtain true PVT values from the observed data, adjustments must be made for the following: (1) elastic stretching of the pipet, (2) thermal contraction of the pipet, and (3) the fraction of the total sample in the capillary and diaphragm cell. Details of these adjustments are given in [1]. Final results are reported by Goodwin, Diller, Weber and Roder [2].

B. *Specific Heat at Constant Volume*

Figure 2 is a cross section of the lower part of the calorimeter cryostat [1]. It is very similar to the PVT cryostat except for the sample container itself, which is a sphere of type 316 stainless steel having a normal volume of 72.35 cm^3 and 1.5 mm walls. Both the inside and outside of the sphere are copper plated to a thickness of 0.2 mm to increase heat conduction. A 100-ohm constantan heater is varnished directly onto the sphere and shielded by the lightweight calorimeter case. During measurements of C_v, the shield, calorimeter, and guard ring are maintained at the same temperature by automatic controllers responding to signals from differential thermocouples.

The sample holder is cooled during loading by means of helium exchange gas in the spaces inside the cold wall and shield. (These two spaces communicate.) This cools the sample holder so that hydrogen may be condensed into it. The sequence of measurements is similar to that of the PVT determinations. For each filling of the calorimeter, the heat capacity as a function of temperature is determined in the usual way, termina-

ting a run at a temperature of about 100°K or a pressure of 300 atmospheres, whichever is reached first. The calorimetric data require adjustment for the same imperfections of apparatus as did the PVT measurements. For the C_v data, these adjustments also include compensating for the work done by the fluid as it expands. Results are given by Younglove and Diller [3, 4] and Goodwin [5].

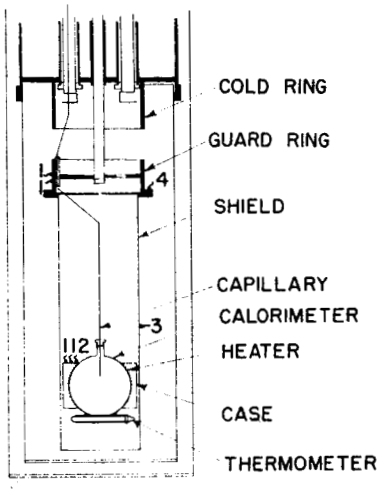

Fig. 2. The C_v Cryostat

CALORIMETER CRYOSTAT

C. *The Velocity and Absorption of Sound in Liquid Parahydrogen*

The velocity and absorption of sound of liquid parahydrogen will be measured from the triple point temperature, approximately 14°K, to 100°K and for pressures from about 10 atmospheres to 340 atmospheres. The method uses ultrasonic pulse methods, originally developed by Pellam in 1946 at MIT and Pinkerton at Cambridge at the same time. A fixed path length is used and the pulse train is long enough so that the signal frequency can be adjusted for constructive interference by comparing the relative phase of the signals of the first and third reflections of the sound wave as registered at the receiving crystal. This is the method of McSkimin of Bell Labs., who works mainly on solids (Figure 3). Attenuation is determined by comparing the amplitudes of the signals received at the first and third reflections.

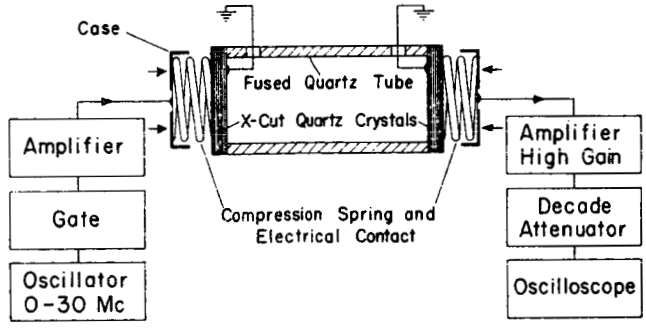

Fig. 3. Diagram of Apparatus to Measure the Velocity of Sound in Fluids

The emitter and receiver of the sound wave are x-cut quartz crystals and the path length is set by an extremely well-made spacer of fused quartz. This is a cylinder with optically flat ends. The length, nominally 6 cm., was measured to 1 part in 150,000 (an average of four measurements) and the ends are parallel to within less than one-half

micron. The variation with temperature and pressure will be accounted for so that the length should be known to 0.001%. Special precautions were taken in design of dimension of the cell and in choice of frequencies so that the velocity of sound values would be accurate to somewhere in the range 0.05% to 0.01%, with greater inaccuracy near the critical point. Accuracy of attenuation will be within a few percent.

D. *Viscosity*

The viscosity of hydrogen is being measured by a method proposed by W. P. Mason [6] in which a cylindrical quartz crystal is caused to undergo torsional oscillations near its resonant frequency. The damping of the oscillations by the fluid surrounding the crystal may be measured in terms of the width of the resonance curve of the crystal. The viscosity-density product of the fluid is related to the bandwidth of the resonance curve by the following expression [7]:

$$[\eta\varrho] = \frac{\pi}{f_0}\left(\frac{M}{S}\right)^2 (\Delta f)^2,$$

where f_0 is the crystal's resonant frequency, M its weight, S its surface area and Δf the bandwidth due to the fluid damping. The measured Δf is adjusted for the damping in vacuum caused by mounting losses and the internal friction of the crystal.

Figure 4 shows a cut-away sketch of the quartz oscillator with a schematic diagram of the measuring circuit. The crystal is suspended at a nodal plane by nylon cords so

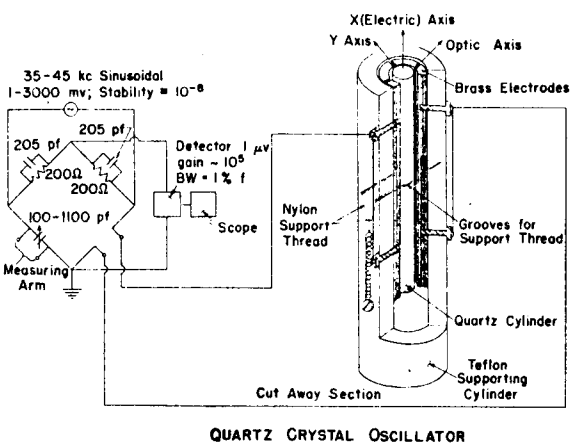

Fig. 4. Apparatus for Measurement of Viscosity

that it can vibrate as freely as possible. The measuring circuit consists essentially of an a. c. bridge for the determination of the crystal's electrical properties and an electronic counter for frequency measurements.

The crystal is shunted by a capacitor to keep the reactance capacitative at frequencies greater than the resonant frequency. The measuring arm of the bridge consists of an accurate decade resistance box in parallel with a variable capacitor. The precision of the measurements depends greatly on the quality of the bridge oscillator and tuned null detector.

The crystal is contained in a sample holder very similar to the PVT piezometer and the same instrumentation is used for the measurement of temperature and pressure. The experimental procedure is also similar to that used for the PVT work except that the previous PVT data are used to compute the density of the hydrogen.

E. *Dielectric Constant of Fluid Hydrogen*

Apparatus is being constructed for the measurement of the dielectric constant of pure parahydrogen in the liquid, gaseous, and super-critical regions. It is planned to measure

the capacitance of a cylindrical capacitor containing hydrogen at a known pressure and temperature. A capacitance bridge, newly on the market, capable of six-figure relative precision, will be used. Since the capacitor contains no dielectric material between its measuring plates, the dielectric constant, ε, of the hydrogen can be obtained as the ratio of capacitor with sample to capacitor evacuated. It is hoped that the quantity $\varepsilon - 1$ can be determined to within a few units in the fourth decimal place.

The previous data on hydrogen can be represented to the precision of the measurements [8] by the Clausius-Mossotti equation,

$$\frac{\varepsilon - 1}{\varepsilon + 2} \cdot \frac{1}{\varrho} = \frac{4\pi}{3M} \alpha,$$

where ε is dielectric constant, ϱ density, M molecular weight, and α the molecular polarizability, assumed constant. It is expected that the present measurements will provide a more sensitive test for departures from the equation that are to be expected.

F. *Engineering Studies*

In addition to measurements of the physical properties of cryogenic fluids such as those just described and many properties of solids which will not be discussed, the Cryogenic Engineering Laboratory has undertaken a number of purely engineering tasks in support of the space program.

Descriptions of two of these will illustrate some of the peculiar problems encountered. The designers of space vehicles early recognized the possibility that difficulty might be expected in venting vapor (without liquid) from a cryogenic propellant tank in the absence of any gravitational field to effect phase separation. To solve the problem they designed centrifugal separators. These required bearings that would operate reliably at low temperatures in either liquid or gas. The Cryogenic Engineering Laboratory had previously made extensive studies of ball bearings running at speeds as high as 9200 RPM submerged in liquid nitrogen [9]. Additional tests in dry gaseous hydrogen showed that these bearings would operate satisfactorily if kept cool by a stream of hydrogen gas [10]. The minimum cooling gas requirement was found to be ¼ to ½ liter per second at 20° to 90°K for a bearing 26 mm OD with a 10 mm bore, supporting a thrust load of about 20 kg.

Another problem associated with the use of cryogenic liquids in space vehicles is the formation of solid when the triple point pressure is reached. It is possible for the solid to block passages or cause malfunction of pumps or valves. The Cryogenic Engineering Laboratory is conducting laboratory studies on such solid formation.

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